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## MEMORANDUM

DATE 12 January 1999

TO David Bennett, WAM, U.S. EPA, Region X

FROM Michelle Turner, Chemist, WESTON, Seattle  
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data  
Laboratory Batch: K9805755  
Site: Duwamish River

WORK ASSIGNMENT NO. 46-35-0JZZ

WORK ORDER NO. 4000-019-038-5200-00

DOC CONTROL NO. 4000-019-038-AAAK

cc Bruce Woods, RAP-WAM, U.S. EPA, Region X  
Dena Hughes, Site Manager, WESTON, Seattle (memo only)  
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of five sediment samples, laboratory batch K9805755, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98354000      98354001      98354006      98354007      98354008

### Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods

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Site Duwamish River

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### 1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met extraction holding time criteria but exceeded analysis holding time criteria as follows:

Sample ID	Date Collected	Date Extracted	Date Analyzed	No of Days
98354000	8/24/98	8/31/98	9/8/98	8 days (analysis)
98354001	8/24/98	8/31/98	9/9/98	9 days (analysis)
98354006	8/24/98	8/31/98	9/9/98	9 days (analysis)
98354007	8/24/98	8/31/98	9/9/98	9 days (analysis)
98354008	8/24/98	8/31/98	9/9/98	9 days (analysis)

Sample results and detection limits were qualified as estimated (UJ/J)

### 2. Detection Limits

Detection limits met project required quantitation limits with the following exceptions

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98354000	n-Butyltin	10	45
98354001	n-Butyltin	10	40
98354007	n-Butyltin	10	30
98354008	n-Butyltin	10	25

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference

### 3 Initial Calibration

A five-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD

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#### 4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. Target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent with the following exceptions:

Sample	Analyte	% Recovery	Associated Samples
CCV4	Tetrabutyltin	126	none
CCV4	Tributyltin	132	none

As no project samples were associated with this continuing calibration, no qualifiers were assigned to sample results.

#### 5. Blanks

##### a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks

##### b) Field Blanks

No field blanks were associated with this SDG.

#### 6. Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory an additional surrogate, Triphenyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below.

Surrogate Compound	Sediment Limits
Tripropyltin	20 - 195%
Triphenyltin	20 - 172%



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Surrogate compound percent recoveries exceeded the QC limits for the following samples.

Sample	Surrogate	Percent Recovery
98354000	Tripropyltin	53
98354007	Tripropyltin	56
K980831-LCS	Tripropyltin	44
K980831-MB	Tripropyltin	28
K980831-MB	Tripropyltin	50

Samples results and detection limits were qualified as estimated (UJ/J) when both surrogate recoveries were outside the QC limits. As only one surrogate was outside the QC limits in each sample, qualifiers were not assigned. Although both surrogates were outside the QC limits in the Method Blank, sample results were acceptable.

7. Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 20 to 164 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions

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LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K980908-LCS	Tetrabutyltin	51	60-130 (P) 20-164 (L)	98354000 through 98354001 98354006 through 98354008
K980908-LCS	Dibutyltin	31	60-130 (P) 20-164 (L)	98354000 through 98354001 98354006 through 98354008
K980908-LCS	n-Butyltin	20	60-130 (P) 20-164 (L)	98354000 through 98354001 98354006 through 98354008

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits.

#### 8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment.

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%

MS/MSD sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following

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Sample	Compound	Percent Recovery	QC Limits
K9805833-007MS (Batch QC)	Dibutyltin	21	30-120 (P) 20-200 (L)
K9805833-007DMS (Batch QC)	Dibutyltin	9	30-120 (P) 20-200 (L)

MS recovery for n-Butyltin was not calculated due to matrix interferences

Relative percent differences (RPD) between the MS and MSD percent recoveries exceeded QC guidelines for the following compounds:

Sample	Compound	RPD
K9805833-007 (Batch QC)	Tetrabutyltin	83
K9805833-007 (Batch QC)	Dibutyltin	47

No action was based solely on MS/MSD data.

9. Field Duplicate Analysis

Samples 98354000 and 98354001 were submitted to the laboratory as "blind" field duplicates. Relative percent differences (RPDs) were less than 25 percent for water and less than 35 percent for sediment samples.

Samples 98354007 and 98354008 were also submitted to the laboratory as "blind" field duplicates. Relative percent differences (RPDs) were less than 25 percent for water and less than 35 percent for sediment samples.

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. A duplicate analysis was performed on Batch QC sample K9805833-010, RPD values were less than 35 percent. The case narrative indicated that the Batch MS recovery for n-Butyltin was not calculated due to matrix interference. The Batch MSD recovery of Dibutyltin was outside the QC limits. As the MS and LCS recoveries for Dibutyltin were



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within QC limits, no action was taken. No other problems were noted in the case narrative.

#### 11. Laboratory Contact

No laboratory contact was required.

#### Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

#### Data Qualifiers

- U - The compound was analyzed for, but was not detected.
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N - Presumptive evidence of presence of material (tentative identification).
- I - Elevated reporting limit due to matrix interference.

## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805755  
**Date Collected:** 8/24/98  
**Date Received:** 8/25/98

## Butyltins

**Sample Name** 98354000 **Units** ug/Kg (ppb)  
**Lab Code** K9805755-001 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/31/98	9/8/98	ND	UJ
Tri-n-butyltin	Method	Butyltins	5	5	8/31/98	9/8/98	36	J
Di-n-butyltin	Method	Butyltins	5	5	8/31/98	9/8/98	27	J
n-Butyltin	Method	Butyltins	45	5	8/31/98	9/8/98	ND	UIJ B

D The MRL is elevated because of matrix interferences and because the sample required diluting  
B The MRL is elevated because of matrix interferences

Approved By



Date

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## Analytical Report

**Service Request:** K9805755  
**Date Collected:** 8/24/98  
**Date Received:** 8/25/98

Units	ug/Kg (ppb)
Basis	Dry

Analyte	Prep	Analysis	MRL	Dilution	Date	Date	Result	Result Notes
	Method	Method		Factor	Extracted	Analyzed		
Tetra-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	UJ
Tri-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	40	J
Di-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	32	J
n-Butyltin	Method	Butyltins	40	5	8/31/98	9/9/98	ND	UIJ B

D	The MRL is elevated because of matrix interferences and because the sample required diluting
B	The MRL is elevated because of matrix interferences

Approved By [Signature] Date 9-22-98  
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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805755  
**Date Collected:** 8/24/98  
**Date Received:** 8/25/98

## Butyltins

Sample Name 98354006 Units ug/Kg (ppb)  
Lab Code K9805755-007 Basis Dry  
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	uJ
Tri-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	6	J
Di-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	uJ
n-Butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	uJ

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805755  
**Date Collected:** 8/24/98  
**Date Received:** 8/25/98

## Butyltins

**Sample Name** 98354007 **Units** ug/Kg (ppb)  
**Lab Code** K9805755-008 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	WJ
Tri-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	26	J
Di-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	WJ
n-Butyltin	Method	Butyltins	30	5	8/31/98	9/9/98	ND	WJ J B

D  
B

The MRL is elevated because of matrix interferences and because the sample required diluting  
The MRL is elevated because of matrix interferences

Approved By



Date

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

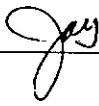
**Service Request:** K9805755  
**Date Collected:** 8/24/98  
**Date Received:** 8/25/98

## Butyltins

**Sample Name** 98354008 **Units** ug/Kg (ppb)  
**Lab Code** K9805755-009 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	ND	WJ
Tri-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	28	J
Di-n-butyltin	Method	Butyltins	5	5	8/31/98	9/9/98	8	J
n-Butyltin	Method	Butyltins	25	5	8/31/98	9/9/98	ND	UIJ B

D The MRL is elevated because of matrix interferences and because the sample required diluting  
B The MRL is elevated because of matrix interferences

Approved By  Date 9-22-98

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